



# **FOOD ENGINEERING AND ENVIRONMENTAL ASPECTS OF MEMBRANE AND OSMOTIC DISTILLATION**

*Thesis of Ph.D. dissertation*

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## 1. INTRODUCTION

Recently, mankind with its wasteful habit risks not only its own future but also the sustainability of Earth's biosphere. As water is essential for the living organisms, the proper quantity and quality of surface and subsurface water resources are indispensable. Industrial activities have changed the conditions of water resources; herewith have impacted directly on the living organisms. By that, it is obvious that one of the crucial challenges of mankind is to discover those novel approaches and processes by which the objectives of sustainable development's principles can be achieved. I believe that these approaches can be found as well as membrane and osmotic distillation going to aid significantly to realize them.

In my PhD thesis I summarize the most characteristic engineering aspects of membrane and osmotic distillation for helping to understand these processes for further development. As a first part of my doctoral work, I demonstrate results about new dynamic measurement technique of liquid entry pressure (LEP) which acts as a key parameter regarding distillate quality. I also compare this new approach to the findings available in the open literature. As a second part I present a new technique to regenerate the wetted pores by vacuum and compare it the usage of elevated temperature regeneration technique. The third part of my PhD work contains results about separation of oil in water emulsion by using vacuum membrane distillation. LEP values of different emulsion composition are also examined regarding the novel dynamic and the other, well-know method. As a fourth part of my thesis some special materials are applied as osmotic agent to improve the process productivity. These materials can be used as food additives or fertilizers after their usage and can be marketed. Finally, sage extract and sour cherry juice are concentrated using integrated membrane process. Valuable compounds of sage extract and sour cherry juice namely total polyphenolic content, total antioxidant activity are followed up to determine the effect of the particulate membrane process steps.

## **2. OBJECTIVES OF THE DISSERTATION**

### **2.1 Examination of membrane pore wetting in case of membrane distillation**

Determination of liquid entry pressure ( $LEP_w$ ) is carried out using a classical, so-called static method and a novel dynamic method. If membrane pores became wetted before an effective pore regeneration technique membrane distillation no longer can be realized. This means a full removal of the moisture from the pores. There is no study in the open literature which focuses on a special technique which is suitable to regenerate the wetted membrane pores quickly and effectively.

#### **Related tasks are:**

- To assemble an experimental rig for  $LEP_w$  determination based on the open literature – static method
- To assemble an experimental rig for  $LEP_w$  determination using novel dynamic method, identify hysteresis properties
- To realize regeneration methods using elevated temperature and vacuum – comparison of these methods

### **2.2 Separation of oil in water emulsion by using vacuum membrane distillation**

If the feed stream contains surface active or organic constituents  $LEP$  value can drastically decrease. In case of these examinations separation of low-concentration of oil in water (O/V) emulsion is realized.

#### **Related tasks are:**

- Change of  $LEP$  value the composition of O/V emulsion using static and dynamic method – comparison of the results
- Separation of O/V emulsion by using vacuum membrane distillation – productivity examination in function of the O/V emulsion's composition
- TOC analysis of the produced distillate to discover separation effectiveness

### **2.3 Enrichment of sage extract's valuable compounds by using integrated membrane process**

Salvia, the largest genus of family Lamiaceae comprises around 900 species worldwide. Salvia species, commonly known as sage, have been used since ancient times for more than sixty different ailments of aches, epilepsy, colds, bronchitis, tuberculosis, hemorrhage, and menstrual disorders. Salvia fruticosa Miller (synonym, Salvia triloba L.) is an important sage species which is native to Mediterranean countries. Based on these, effective and gentle preservation techniques need to be improved and integrated membrane processes seem to be a proper solution.

#### **Related tasks are:**

- To carry out hot water extraction of dried Greek sage (*Salvia Fruticosa* Miller) leaves
- Pre-filtration of raw extract by microfiltration and determination of critical and limiting parameters regarding permeate flux and transmembrane pressure difference
- Pre-concentration of pre-filtered sage extract by using reverse osmosis, determination of critical parameters
- Final concentration of sage extract by using osmotic distillation
- To follow up the total polyphenolic content, total antioxidant capacity of raw sage extract, pre-concentrated sage, and final concentrate using spectrophotometric analytics and to determine individual polyphenol compounds by using high-performance liquid chromatography (HPLC)

### **2.4 Examination of special osmotic agents**

However, osmotic distillation is a promising concentrating technique, some limitation still exist such as relative low distillate flux and proper management of exhausted (diluted) osmotic agent. In this part of the thesis saturated solutions prepared from several organic and inorganic salts are tried as osmotic solution to improve process performance. The exhausted osmotic solutions can be optionally marketed as food additives or fertilizers.

**Related tasks are:**

- To examine new osmotic agents to improve distillate flux relative to pure water as feed
- To performe 2<sup>p</sup> factor analysis for the most effective osmotic agent regarding operation parameter feed temperature, temperature difference between feed and osmotic solution stream; saturation condition of the osmotic solution

**2.5 Concentration of sour cherry juice by using integrated membrane process – using new osmotic solution**

After pressing sour cherry fruits, raw juice is pre-filtered using cloth filter and microfiltration. Reverse osmosis is applied as pre-concentration step and finally, osmotic distillation is performed using four different new osmotic solutions to reach high concentration rate.

**Related tasks are:**

- Microfiltration of sour cherry juice, to determine the resistances of the microfiltration process
- Pre-concentration using two different reverse osmosis configurations
- Final concentration by using four different osmotic solutions
- To examine mass transfer phenomenon of the most effective osmotic solution
- Examination of the total polyphenol content and total antioxidant capacity of the sour cherry juice in the different steps of the used integrated membrane process

### 3. MATERIALS AND METHODS

#### 3.1 Examination of membrane pore wetting in case of membrane distillation

LEP<sub>w</sub> value of Durapore™ GVPH flat sheet membrane (Merck Millipore Inc., Billerica, USA) was determined by using static and novel dynamic method in vacuum membrane distillation (VMD) configuration. Furthermore, the novel dynamic method was also applied to determine LEP<sub>w</sub> MD 020 CP 2N hollow fiber module (Microdyn-Nadir GmbH, Wiesbaden, Germany) in direct contact membrane distillation (DCMD) configuration. The examination of the membranes was accomplished in two steps: first, LEP<sub>w</sub> value then hysteresis curves of the wetted membranes were determined. In DCMD configuration there was no temperature difference between feed and distillate side streams, thereby, effect on the LEP<sub>w</sub> of the flowing streams can be investigated. In case of VMD overpressure on the feed side and vacuum on the distillate side was applied to evolve the LEP<sub>w</sub>. Every 5 minutes feed stream hydraulic overpressure was increased with 0.2 bars. In a certain moment liquid stream appeared on the distillate side and the hysteresis curve can be depicted by measuring the change of mass in time.

#### 3.2 Regeneration techniques of the wetted pores

Both Durapore™ GVPH flat sheet membrane (Merck Millipore Inc., Billerica, USA) and MD 020 CP 2N hollow fiber module (Microdyn-Nadir GmbH, Wiesbaden, Germany) were examined in this section. First method means the usage of elevated temperature (50°C) and second method means the usage of vacuum for removing moisture from the pores. For improving regeneration methods, ethanol was rinsed through the system in case of both techniques. To determine the effectiveness of the regeneration method, DCMD and VMD processes were carried out using salty water. If the distillate conductivity was measured lower than 0.01 mScm<sup>-1</sup>, the regeneration procedure was successful.

In case of usage of vacuum, vacuum was applied three different ways: only from the feed stream side, only from the distillate stream side, from both stream sides. These were carried out with or without ethanol rinsing procedure.

#### 3.3 Separation of oil in water emulsion by using vacuum membrane distillation

Determination of Durapore™ GVPH flat sheet membrane's LEP value regarding different oil content of oil in water emulsion (0-3200 ppm) was carried out. Separation of such oil in water emulsion was also realized applying experimental rig depicted on Figure 1.

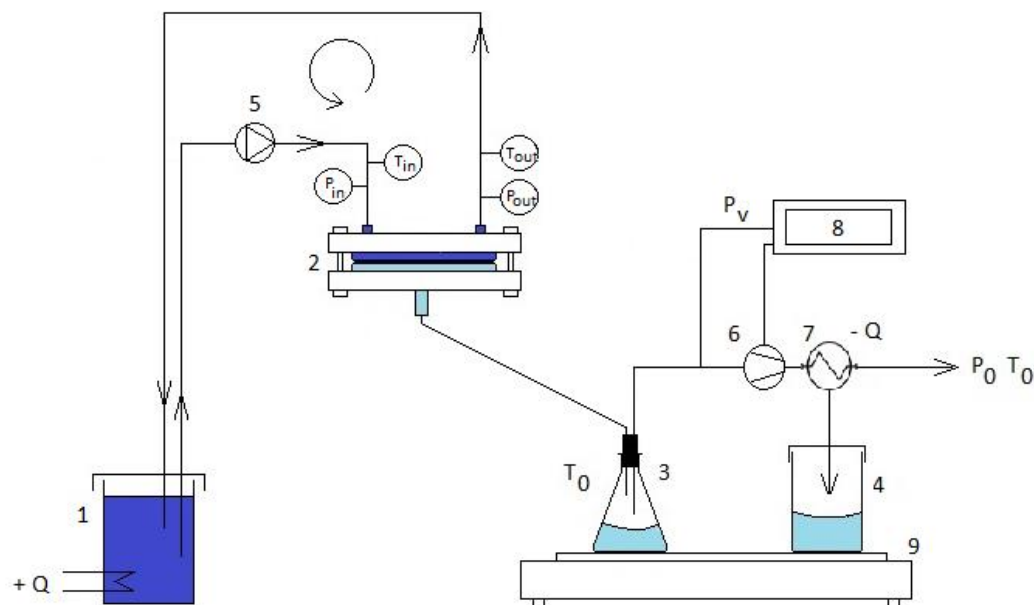


Figure 1 VMD configuration for oil in water emulsion separation (1. for oil in water emulsion, 2. flat sheet membrane in a sandwich module, 3. pre-condense container, 4. condense container, 5. peristaltic pump, 6. vacuum pump, 7. condensator, 8. digital display of vacuum level, 9. digital scale)

### 3.4 Enrichment of sage extract's valuable compounds by using integrated membrane process

The integrated membrane process contained three steps: a microfiltration, a reverse osmosis and osmotic distillation procedure (MF-RO-OD). Fig. 2 illustrates the schematic flow-chart of the sage extract concentration by using the suggested integrated membrane process. Firstly, MF was applied to clarify the initial extract. The permeate stream of the MF process was collected and used for the following steps. After the MF treatment, RO was carried out to pre-concentrate the clarified sage extract and finally, OD was applied to reach the required concentration level.



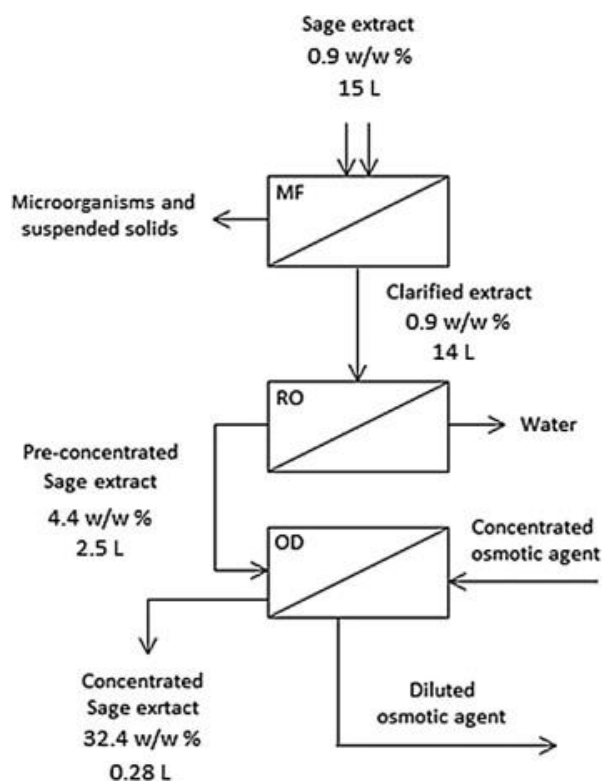


Figure 2 Schematic flow-chart of the suggested integrated membrane process (MF-RO-OD) for concentration sage extract

Total phenolic content (TPC) was analyzed by using the method described by Dincer et al. For this purpose, 0.5 mL of the sample was treated with 2.5 mL of 0.2 N Folin–Ciocalteu reagent and 2 mL of  $\text{Na}_2\text{CO}_3$  (75 g/L). The mixture was incubated at 50 °C for 5 min and then immediately cooled. The absorbance of the final solution was recorded with a spectrophotometer (Shimadzu UV–Vis 160A, Japan) at 760 nm wavelength against deionized water. The results were expressed as gallic acid equivalent (mg GAE/mL extract).

To determine the total flavonoid content (TFC), 2.5 mL distilled water and 150  $\mu\text{L}$  of 5%  $\text{NaNO}_2$  solution was added into a 0.5 mL sample then they were allowed to stand for 5 min after intensive shaking. Then 300  $\mu\text{L}$  of 10%  $\text{AlCl}_3$  solution was added to the solution and allowed to stand for 5 min again. Further 1 mL of 1 M NaOH was added and final volume was made up to 5 mL with distilled water. Sample absorbance was measured at 510 nm by using a spectrophotometer (Shimadzu UV–Vis 160A, Japan) against deionized water. The results were expressed as (+) catechin equivalent (mg CE/mL extract).

The antioxidant activity of the samples was analyzed by using DPPH assay according to the procedure of Dincer et al. 100  $\mu$ L of the diluted sample (prepared at 4 different concentrations which provided 10–90% inhibition) was added into 4 mL of freshly prepared DPPH (2,2 Diphenyl-1-picrylhydrazyl radical) solution ( $6 \times 10^{-5}$  M). The mixtures were shaken and kept in dark at room temperature for 30 min. Absorbance values of the final solutions were recorded at 516 nm using a spectrophotometer (Shimadzu UV–Vis 160A, Japan) against the control (deionized water instead extract in DPPH solution) solution.

The chromatographic separation was performed on a solvent delivery system (20AD, Shimadzu, Japan) coupled with an auto-sampler (SIL-20A Prominence, Shimadzu, Japan), column (LiChroCART® 250-4 250 mm  $\times$  4 mm 5  $\mu$ m Nucleosil® 100-5C 18) and a guard column LiChroCART® 4-4 Nucleosil® 5C 18), maintained at 30 °C in a column oven (CTO-20AC, Shimadzu, Japan). Individual peaks were detected by SPD-M20A Diode Array Detector (Shimadzu, Japan) which is controlled by LC solution software. The mixture of water/acetic acid/methanol (88/2/10, v/v/v) (solvent A) and methanol/acetic acid/water (90/2/8, v/v/v)(solvent B) was used as mobile phase in the following gradient elution at 0.9 mLmin<sup>-1</sup> flow rate: initial A:B 100:0, at 15 min A:B 85:15, at 25 min A:B 50:50, at 35 min A:B 30:70, at 50 min A:B 25:75, and at 55 min A:B 100:0. For the quantification of the individual phenolic compounds the following preparation was applied. 20  $\mu$ L of sample was injected to the column and the separated peaks were monitored at their wavelengths at maximum absorbance (280 (hesperetin), 320 (caffeic acid, p-coumaric acid, ferulic acid, rosmarinic acid), 335 (apigenin), 350 (rutin), nm). Individual peaks were identified by comparison of the retention time and spectrum pattern of their authentic standards. They were also validated by spiking. Quantification was performed by plotting off our different external standards concentrations versus peak areas.

### 3.5 Examination of special osmotic agents

MD 020 CP 2N (Microdyn-Nadir GmbH, Wiesbaden, Germany) hollow fibre polypropylene porous membrane with 0.2  $\mu$ m pore size was applied. The useful membrane area was 0.2 m<sup>2</sup>. 1000 g saturated solutions of CH<sub>3</sub>COOK, NH<sub>4</sub>NO<sub>3</sub>, Ca(NO<sub>3</sub>)<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub>, CaCl<sub>2</sub>, NaNO<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, KNO<sub>3</sub>, NaCl were examined at 30°C. After pre-treatments osmotic agent achieved the highest distillate flux was applied for further examinations. For 2<sup>p</sup> factor analysis the following operation factors and examination range were chosen: T<sub>feed</sub>=32-42°C,  $\Delta$ T=5-15°C, saturation rate 25-100% range.

### 3.6 Concentration of sour cherry juice by using integrated membrane process – using new osmotic solutions

Mixture of five Hungarian cultivars of sour cherries (“Érdi bőtermő”, “Debreceni bőtermő”, “Oblacsinszka”, “Pipacs I”, “Újfehértói fürtös”) was used as feed for the experiments. After pressing, the initial total soluble solid (TSS) of juice was 9.2 °Brix. Sour cherry juice was clarified using cross-flow microfiltration. Tubular ceramic membrane was applied with 0.45 µm pore size (Millipore, Billerica, MA, USA) at 25 °C and 1.5 bar transmembrane pressure difference. Furthermore, reverse osmosis was applied to pre-concentrate the clarified juice using a spiral wound membrane module (MFT Cologne, CD1TYP3, Cologne, Germany) with 98% of NaCl retention at 30 °C and 30 bar transmembrane pressure difference. 20 °Brix of TSS was reached.

MD 020 CP 2N (Microdyn-Nadir GmbH, Wiesbaden, Germany) hollow fibre polypropylene porous membrane with 0.2 µm pore size was applied. The useful membrane area was 0.2 m<sup>2</sup>. Water activity values of the sour cherry juice and osmotic agents were determined using Novasina LabMASTER-a<sub>w</sub> (Novasina AG, Lachen, Switzerland) equipment. Water activity values were determined at different concentrations up to the final concentration of sour cherry juice and the saturation concentration of the examined osmotic agents.

Microbiological analysis included enumeration of yeast, mould and total bacterial counts in the samples. Triptone Glucose Extract Agar (TGEA) and Dichloran Rose Bengal Chloramphenicol Agar, 1.00466, Merck (DRBC) have been used for TBC and yeast and mould enumeration respectively. Spread plate (Microbiology of food and animal feeding stuffs. Horizontal method for the enumeration of the yeast and mould, colony count technique in products with a<sub>w</sub>=0.95, ISO 21527,2008) and pour plate (Microbiology –general guidance for the enumeration of microorganisms colony count technique at 30°C, ISO 4833, 2nd edition 1991) methods were used for the enumeration of TBC and yeast moulds, respectively.

Antioxidant power was measured with the FRAP (ferric reducing ability of plasma) method (Benzie and Strain, 1996). The antioxidant power was calculated from a standard curve obtained by different concentrations of ascorbic acid.

Total amount of soluble phenols was determined using Folin-Ciocalteu’s method and reagent according to the method of Singleton and Rossi (1965). The content of soluble phenols was calculated from a standard curve obtained by different concentrations of gallic acid.

## 4. RESULTS

My thesis focuses on two main topics: identification of LEP value and the regeneration of the wetted membranes and based on these, treatment of oil-in-water emulsion to produce clear high purity water. Another main topic is to intensify osmotic distillation process applying new osmotic solutions which later can be sold as product without supplement regeneration process. Finally, coupled membrane process containing microfiltration (MF), reverse osmosis (RO) and osmotic distillation has been applied to concentrate sage extract and sour cherry juice.

Regarding membrane distillation, a new approach has been developed to determine one of the critical parameters in MD processes, the so-called liquid entry pressure (LEP). This approach possesses advantages compared to the classical, „static” method, because it is imitating a real process run. From this point of view operators can extract information which can be more crucial regarding distillate quality and process efficiency as well. Based on these results separation of an oil-in-water emulsion is practically feasible, however, a critical concentration of the dispersed oil exists. This limit can be only overcome with new-type of membranes which have to be developed in the near future. These membranes are still under construction, but in small-scale application have already been achieved.

Concentrating sage extract can be achieved using coupled process. MF-RO-OD coupled process is suggested in my thesis which can use four effective concentrations of the extract. However, significant valuable compound loss is occurred along with the RO process. In case of osmotic distillation negligible valuable compound loss is determined.

Applying osmotic distillation with  $\text{CH}_3\text{COOK}$  solution is a promising option. This salt is very soluble, thus, a solution with very low vapor pressure can be reachable, it can be perfectly used for effective osmotic distillation processes. Furthermore, this compound is a well-known food additive, thus non-toxic, it can be applied in liquid food concentration tasks. The results have presented that using  $\text{CH}_3\text{COOK}$  is more effective than the classical  $\text{CaCl}_2$ , however, the juice suffers valuable compound loss along the process. This can origin from the ambient physical effect such as oxidation with oxygene in the air, or photochemical effect, or even temperature. To find the right answer more experiments are required.

## 5. NOVEL SCIENTIFIC RESULTS

I. **I have successfully developed a novel dynamic method for liquid entry pressure (LEP<sub>w</sub>) determination in DCMD and VMD configurations.** I have proved that novel dynamic method is a proper method for LEP<sub>w</sub> determination. I have also proved that in case of VMD configuration LEP<sub>w</sub> value determined by dynamic method shows lower value compared to static method. However, LEP<sub>w</sub> value determined by dynamic method claims more rigorous engineering implementation aspects, but results higher product quality.

II. **I have successfully implemented a new vacuum regeneration procedure for flat sheet membranes in sandwich modules.** This procedure significantly facilitates the removal of moisture from the wetted membrane pores without disassembling the unit.

III. **I have proved that by using Durapore™ GVPH membrane in VMD configuration there is no effect on distillate flux of oil in water emulsion's oil concentration up to a certain composition by which a spontaneous wetting occurs.** This value has been determined as 250 ppm under the examined conditions. Furthermore, I have also proved that LEP values of the O/V emulsion determined by dynamic method shows lower values compared to static method. This phenomenon has been also found and presented already in novel scientific result I., as an important finding.

IV. I have examined barely or even never applied inorganic and organic salts as osmotic agent for improving osmotic distillation process performance. **I have proved that saturated CH<sub>3</sub>COOK solution is the most effective osmotic solution among them.** After performing 2<sup>p</sup> factor analysis the following regression model has been determined.

$$J = 0,8908 + 0,6019 \frac{C(\%) - 62,5}{37,5} + 0,1704 \frac{T(^{\circ}C) - 37}{5}$$

which is valid at T<sub>feed</sub>=32-42°C, ΔT=5-15°C, saturation rate 25-100% range.

V. **I have determined that** along with the concentration of sour cherry juice by using integrated membrane process contained microfiltration, reverse osmosis and osmotic distillation steps, **the total polyphenolic content has decreased significantly during both spiral wound and flat-sheet membrane reverse osmosis processes.** Osmotic distillation processes performed by four different osmotic agents have also resulted significantly decreased total polyphenolic content. In case of total

antioxidant capacity content I have proved that both spiral wound and flat sheet membranes can successfully pre-concentrated the sour cherry juice without significant degradation, however, osmotic distillation processes performed by four different osmotic agents have also resulted significantly decreased total antioxidant capacity. Furthermore, **I have determined that the degradation of valuable compounds has been less pronounced by the usage of  $\text{CH}_3\text{COOK}$**  which phenomenon can be explained by the less contact time for oxidative stress factors (light, temperature, oxygen).

VI. I have achieved a multistep, integrated membrane process for enrichment of sage (*Salvia fruticosa* Miller) extract's valuable compounds by using integrated membrane process (MD-RO-OD). **I have proved that the initial 0.9 m/m% total soluble solid content can be increased up to 32.4 m/m% by using microfiltration-reverse osmosis-osmotic distillation integrated process. I have also concluded that the reverse osmosis step can retain valuable compounds 60%, however, osmotic distillation can retain more than 90%.**

## 6. CONCLUSIONS, SUGGESTIONS

- I. LEP value determined by using dynamic method shows more “realistic” situation compared to static method can be found in the open literature. As a first step of a design and implementation activity, the adequate determination of this parameter is essential, and the novel dynamic method is suitable to realize this.
- II. Applying vacuum to regenerate wetted membrane pores can be an appropriate technique, because the long and expensive disassembly can be avoided. However, a special module configuration with transparent material could help to discover the internal structure and dead-zones of the module where moisture can be piled up regarding vacuum sucking. By this, more effective methods can be realized.
- III. Vacuum membrane distillation process is suitable to separate oil in water emulsions, however, the modul structure is a crucial factor. Another key parameter is the critical oil concentration, thus, it is recommended to operate the system in continuous mode under this limitation factor. Further experimental investigations are suggested to analyse such systems.
- IV. Concentration of sage extract by using membrane processes is a promising option, however, critical technique is the reverse osmosis by which significant valuable compound loss can occur. It is recommended to investigate different membranes and operation parameters by which these phenomena can be lowered as much as possible.
- V. Saturated  $\text{CH}_3\text{COOK}$  solution is an excellent alternative to improve osmotic distillation process. Food industrial usage can be managed; therefore, the regeneration of the exhausted osmotic solution can be avoided.
- VI. In the examined operation parameter range there is no significant effect on the distillate flux of the temperature difference between the feed solution and the osmotic solution. It is recommended to extend the examination range widely for further investigation.
- VII. Integrated membrane process (MF-RO-OD) is suitable to concentrate sour cherry juice; however, appropriate reverse osmosis membranes need to be chosen. Another important task is to determine precisely which physical or chemical effect results the significant valuable compound loss during osmotic distillation concentration procedure and how it could be eliminated.

## LIST OF PUBLICATIONS

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